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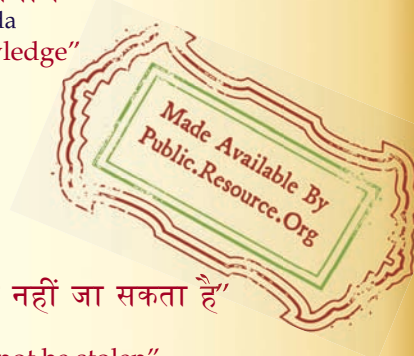
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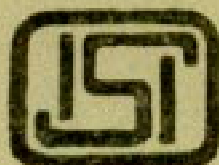
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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SOLDERS FOR USE IN GOLDWARE

PART I DETERMINATION OF GOLD, SILVER AND COPPER

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METHODS OF CHEMICAL ANALYSIS OF SOLDERS FOR USE IN GOLDWARE

PART I DETERMINATION OF GOLD, SILVER AND COPPER

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SOLDERS FOR USE IN GOLDWARE

PART I DETERMINATION OF GOLD, SILVER AND COPPER

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 March 1974, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Nine grades of gold solders have been specified in IS:3095-1966*. In order to determine correctly the composition of different gold solders, methods of analysis are given in this standard. It covers the analysis of gold, silver and copper in gold solders. The methods for the determination of cadmium and zinc in gold solders are being investigated and will be added later.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960†.

1. SCOPE

1.1 This standard covers the methods for determination of gold, silver and copper in solders for use in goldwares as specified in IS:3095-1966*.

2. QUALITY OF REAGENTS

2.1 Unless otherwise specified, pure chemicals and distilled water (see IS:1070-1960‡) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for solders for use in goldware.

†Rules for rounding off numerical values (*revised*).

‡Specification for water, distilled quality (*revised*).

3. PREPARATION OF TEST SAMPLE

3.1 Roll the sample to very thin size, cut and mix up.

4. DETERMINATION OF GOLD BY THE FIRE ASSAY METHOD

4.0 Gold may be determined by the following fire assay method or during the determination of copper (see 6.4.1).

4.1 Outline of the Method — After the addition of definite proportion of silver, the sample is cupelled along with sufficient amount of lead to remove copper and zinc. The resulting gold-silver button is flattened, parted and gold cornet weighed.

4.2 Apparatus

4.2.1 Cupellation Furnace — It is a muffle type furnace provided with inlets and outlets for air so that a current of air, preferably preheated, may constantly pass through it. The muffle shall be capable of being heated uniformly up to a temperature of 1 100°C.

4.2.2 Assay Balance — of highest precision capable of carrying 1 or 2 g in each pan and sensitive to 0.01 mg. The balance should be so adjusted that one division of the scale traversed by the pointer corresponds to 0.05 mg.

4.2.3 Gold Assay Weights — shall be of the highest accuracy and made of platinum-iridium. Figures shall be stamped on the weights to denote the number of half-milligrams or milliemes contained in the weight, the 0.5 g weight shall be stamped '1000'.

4.2.4 Cupels — made of bone-ash or magnesia.

4.2.5 Rolling Mill — a small jewellers' rolling mill.

4.2.6 Rolling Pliers — a pair of steel pliers with smooth concave chops, the diameter of the cavity in the chops being about 10 mm.

4.2.7 Parting Tray — a number of small thimblelike perforated cups of platinum or silicon supported in a frame of the same material.

4.2.8 Hammer and Anvil — with bright and clean striking faces.

4.2.9 Trays — of various forms for keeping the assay-pieces, buttons, rolls, fillets, cornets, etc.

4.2.10 Tongs and Forceps — of various forms, for charging the cupels and handling assay-pieces, etc.

4.3 Reagents

4.3.1 Check Gold — of fineness 999.9.

4.3.2 Silver — free from gold.

4.3.3 Lead Foil — of pure lead free from silver.

4.3.4 Parting Acid No. 1 — Dilute nitric acid (2:3) (*v/v*) containing a trace of silver nitrate.

4.3.5 Parting Acid No. 2 — Dilute nitric acid (5:2) (*v/v*) containing a trace of silver nitrate.

4.4 Procedure

4.4.1 For 14 Carat and 12 Carat Samples

4.4.1.1 Weighing of the sample — Weigh accurately 0.25 g (or 500 milligrammes gold assay weight) of the sample within ± 0.4 milligrammes (note and allow for the correction in the final weighing of the gold cornet). Add to it 0.25 g (or 500 milligrammes gold assay weight) of check gold of known fineness weighed accurately within ± 0.4 milligrammes. Note and allow for this correction in the final weighing.

4.4.1.2 Preparation of the assay piece — Add 1 g of pure silver to the sample and wrap in a lead foil (weight about 5 g) and squeeze in the balling pliers to form a small ball. The assay piece shall now be considered ready for charging.

4.4.2 For 9 Carat Gold Sample

4.4.2.1 Weighing of the sample — Weigh accurately 0.5 g (or 1 000 milligrammes gold assay weight) of the sample within ± 0.04 milligrammes. Note and allow for this correction in the final weighing of the gold cornet.

4.4.2.2 Preparation of assay sample — Dissolve the sample in the parting acid No. 1 by heating. When the action stops, allow it to cool and filter using No. 1 filter paper. Wash with hot water, several times. Ignite the paper at 800°C. Transfer the residue to a lead foil (mass about 5 g). Add to it 0.4 g pure silver and squeeze in the balling pliers to form a small ball.

4.4.3 Preliminary Assay — If the approximate composition of the sample of gold alloy is not known, it is necessary to make a preliminary assay. For this purpose, weigh 2 lots of 100 mg of the alloy, and 300 mg of silver to one lot and then wrap each lot separately in 3 g of lead. Cupel both side by side. Flatten the button containing the added silver and boil in 15 ml of parting acid No. 2 for 15 minutes, wash the resulting gold, dry and heat to redness and weigh. The mass in milligrams gives directly the percentage of gold and the mass in milligrams of the other button gives the percentage of gold and silver together; the difference between the two gives the percentage of silver. The remainder will be base metal. When the approximate composition of the alloy has been ascertained, determine the amount of silver to be added for assay.

4.4.4 Cupellation — Arrange the cupels carefully in the muffle, preferably on a removable tray. When the cupels have attained the furnace temperature of about 1 000°C place in them the assay pieces each in its proper cupel, by means of a pair of long cupellation tongs. The charging in should be done carefully, but as rapidly as possible, so as not to cool the muffle unduly. Close the muffle door and allow the cupellation to proceed for 20 to 30 minutes depending on the amount of lead used, the temperature being raised to about 1 050°C towards the end. The end of the cupellation is shown by the appearance of bright globules of gold-silver alloy. The cupels may be withdrawn while the temperature comes down to approximately 850°C in the furnace until the buttons are solidified and then removed from the furnace.

4.4.5 Preparing the Assay-Piece for Parting

4.4.5.1 Remove the buttons from the cupels by means of a pair of forceps and clean with a stiff brush. Flatten the buttons on a polished anvil with a polished hammer. Anneal the flattened buttons at about 800°C and pass in succession through the rolls to form elongated fillets of thickness 0.25 mm and number them serially. After being rolled, anneal to soften them and then separately roll up between the finger and thumb into a 'cornet' or spiral; making the lower side of the bottom the outer face of the cornet.

4.4.5.2 Place the cornets in the respective cups of the parting tray and immerse the entire tray in parting acid No. 1 at a temperature of 90 to 92°C and boil for 25 to 30 minutes or until no more nitrous fumes are observed. Take out the tray and drain from acid liquor, then wash by dipping in and out of a vessel of hot distilled water, drain again and immerse in a second lot of boiling parting acid No. 2. Boil the cornets in this way for 20 to 25 minutes, drain and wash thoroughly. Dry the tray with the cornets by gently heating and then anneal in a muffle heated to 800°C, for about 5 minutes. The cornets which before annealing are of a dull brown colour and friable now assume a bright gold yellow colour, diminish in size and harden. The cornets when cold are ready for weighing.

4.4.6 Check Assays

4.4.6.1 Certain losses and gains are incurred in the various operations of assay due mainly to losses of gold and retention of silver by cornet. The net sum of these losses and gains is called 'surcharge'. The surcharge shall be determined by means of check-assays or 'checks' of similar composition as sample or samples being assayed. The checks shall be made according to the known composition of the sample or from the data obtained by preliminary assay according to 4.4.3, if the approximate composition of the sample is not known. Check-gold shall always be used for the preparation of the checks and taken as the standard against which all assays shall be compared.

NOTE — If the negative surcharge is obtained, the assay shall be repeated.

4.4.6.2 The checks shall be subjected to the same operations side by side and under identical conditions with the assay-pieces. The number of checks shall be not less than two for each group of assays and shall be positioned evenly in the group.

4.4.7 Method of Reporting

4.4.7.1 For 14 carat and 12 carat samples — Weigh the cornets accurately in the assay balance in tenths of milliims. Apply proportionate correction and surcharge previously determined to the mass thus obtained and deduct the amount of gold added. Record the mean value of the duplicate test results provided the difference is not more than ± 0.04 milliemes. Report this mean value per milliemes by multiplying by 2.

4.4.7.2 For 9 carat — Weigh the cornet accurately in the assay balance in the tenths of milliemes and apply proportionate correction and surcharge previously determined. Record the mean value of the duplicate test results provided the difference is not more than ± 0.4 milliemes.

4.4.7.3 If the difference does not show this agreement, repeat the test in duplicate.

4.4.7.4 Out of the four results thus obtained, locate the most divergent result. Observe the difference (D) between the most divergent result and the mean of the other three close results, that is, the results where the difference between the lowest and the highest value is less than or equal to 0.8 milliemes. Report the results as explained below:

Sl No.	Difference (D)	Method of Reporting	Examples	
			Results Obtained (in milliemes)	Result to be Reported (in milliemes)
a)	D is ≤ 0.4 milliemes	Mean of the four results to be re- ported	948.2, 948.7, 948.4 & 948.3	948.4
b)	D is over 0.4 milli- emes	Mean of the three close results to be reported	911.3, 911.8, 911.4 & 912.2 (Most divergent)	911.5

4.4.7.5 If after locating the most divergent result, it is found that the difference between the lowest and the highest results among the remaining three values is over 0.8 milliemes, all the four results should be individually reported.

Example: 901.5, 900.1, 903.1 and 901.2.

4.4.7.6 When it is not possible to decide the most divergent result, as shown in the examples, all the four results should be individually reported.

Example 1: 946·9, 946·2, 946·2 and 946·9 millimetres.

Example 2: 920·7, 921·3, 919·9 and 920·5 millimetres.

5. DETERMINATION OF SILVER (BY SILVER CHLORIDE PRECIPITATION) METHOD

5.1 Outline of the Method — The sample is first treated with dilute nitric acid and then the residue treated with nitric acid-hydrochloric acid for complete dissolution. Silver is precipitated as silver chloride.

5.2 Reagents

5.2.1 Dilute Nitric Acid — 1 : 1 (v/v).

5.2.2 Dilute Hydrochloric Acid — 1 : 1 (v/v).

5.2.3 Dilute Ammonia — 1 : 10.

5.3 Procedure

5.3.1 Transfer an accurately weighed 0·5 g of test sample to a 500-ml beaker. Treat with 15 ml of dilute nitric acid, cover with watch-glass and boil carefully for 20 minutes. Decant the solution, washing with hot water, in a second beaker.

5.3.2 To the residue in the original beaker, add 15 ml of dilute nitric acid and warm slowly. Add 45 ml of dilute hydrochloric acid, cover with watch-glass and keep on a water bath to dissolve the sample completely. Transfer the contents to the second beaker reserved under

5.3.1. Dilute with 250 ml of water and boil for 2 to 3 minutes and allow to settle for about two hours. Filter off silver chloride through Whatman filter paper No. 30 or its equivalent and reserve the filtrate for copper determination. Dissolve the silver chloride in dilute ammonia, filter through the crucible, wash the crucible with dilute ammonia and acidify the filtrate with dilute nitric acid. Boil for 2 to 3 minutes and allow the silver chloride precipitate to settle for overnight away from direct sun light. Filter through tared sintered glass crucible of porosity No. 3. Dry the precipitate at 120 to 130°C for about 1 hour to constant mass.

5.4 Calculation

$$\text{Silver, percent} = \frac{A \times 75.26}{B}$$

where

A = mass in g of silver chloride, and

B = mass in g of the sample taken.

6. DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD

6.1 Outline of the Method — From the solution reserved under 5.3.2, gold is removed by sulphur dioxide and copper in the filtrate is deposited electrolytically and weighed.

6.2 Apparatus — same as given in IS:4667 (Part I)-1968*.

6.3 Reagents

6.3.1 Concentrated Hydrochloric Acid — sp gr 1.16 (conforming to IS:265-1964†).

6.3.2 Concentrated Nitric Acid — sp gr 1.42 (conforming to IS:264-1968‡).

6.3.3 Concentrated Sulphuric Acid — sp gr 1.84 (conforming to IS:266-1961§).

6.3.4 Sulphur Dioxide — gas.

6.3.5 Urea — solid.

6.3.6 Ethanol or Methanol — 95 percent (v/v).

6.4 Procedure

6.4.1 Evaporate the filtrate reserved under 5.3.2 to syrupy consistency and add 15 ml of concentrated hydrochloric acid, dilute to about 200 ml and heat to boiling. Add 50 ml of sulphurous acid to precipitate gold. Stir for a few minutes and allow to stand on a hot-plate or water bath for 2 hours. Allow it to settle overnight and filter through Whatman filter paper No. 42 or its equivalent. Wash with hot water till the filtrate is free from chloride. Preserve the filtrate and washings. Dry the paper and then ignite it at 800°C. Weigh for the determination of gold.

6.4.2 To the filtrate and washings obtained under 6.4.1 add 5 ml of concentrated sulphuric acid and evaporate to fumes, cool, dilute to 200 ml and add 5 ml of concentrated nitric acid. Add 2 g of urea, insert the electrodes, the cathode having been accurately weighed; cover with a pair of split cover-glass and electrolyze for 16 hours at a current density 0.6 A/dm² (at this current density, the electrolysis is conveniently carried on overnight), or at a current density of 4 A/dm² for a short period (about one hour). In the latter case, one of the types of rotating forms of electrodes generally available may be used. When the solution

*Methods of chemical analysis of silver-copper brazing alloys: Part I Analysis for silver and copper.

†Specification for hydrochloric acid (*revised*).

‡Specification for nitric acid (*first revision*).

§Specification for sulphuric acid (*revised*).

appears colourless reduce the current density to 0.3 A/dm² and continue electrolysis until the deposition of copper is complete as indicated by absence of plating on the new surface of the electrode obtained by lowering the electrode.

6.4.3 Without interrupting the current, lower the beaker or raise the electrode, as the case may be, slowly while rinsing the electrodes with water and collecting the washing in the electrolyte. Remove the cathode quickly, rinse it with water in a beaker and then dip it in two successive baths of ethanol or methanol. Dry the cathode in an air-oven at 110°C for 3 to 5 minutes, cool and reweigh the cathode. The difference in weighings gives the mass of copper deposited.

6.5 Calculation

$$\text{Copper, percent} = \frac{A}{B} \times 100$$

where

A = mass in g of copper deposited, and

B = mass in g of the sample taken.

INDIAN STANDARDS

ON

PRECIOUS METALS

IS:

639-1965	Gold leaf (<i>revised</i>)
1417-1969	Grades of gold and gold alloys (<i>first revision</i>)
1953-1962	Methods for chemical analysis of silver anodes
2112-1962	Grades of silver and silver alloys
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